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Effect of step-quenching on microstructure of aluminum alloy 7055

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Abstract: The effect of step-quenching on the microstructure of aluminum alloy 7055 after artificial aging was studied by hardness testing and transmission electron microscopy (TEM). Step-quenching leads to decomposition of solid solution and heterogeneous precipitation of equilibrium phase mainly on dispersoids and at grain boundaries; thus lower hardness after aging. Prolonging isothermal holding at 415 ℃ results in coarser and more spaced η phase particles at grain boundaries with wider precipitates free zone, and lower density of larger η' hardening precipitates inside grains after aging. Isothermal holding at 355 ℃ results in heterogeneous precipitation of η phase both on dispersoids and at grain boundaries. Isothermal holding at 235 ℃ results in heterogeneous precipitation of η phase first, and then S phase. Precipitates free zones are created around these coarse η and S phase particles after aging. Prolonging isothermal holding at these two temperatures leads to fewer η' hardening precipitates inside grains, larger and more spaced η phase particles at grain boundaries and wider grain boundary precipitates free zone after aging.
Key words: quenching; aluminum alloy 7055; heterogeneous precipitation; η phase; precipitates free zone

1 Introduction

Al-Zn-Mg-Cu aluminum alloys are aging-hardenable. The high strength is obtained by formation of fine precipitates due to decomposition of super-saturated solid solution. These fine precipitates are generally developed by homogeneous nucleation during aging treatments, so many investigations were focused on this topic[1−3]. In order to attain good aging hardening effect, highly super-saturated solid solution is essential. As these alloys are quenching sensitive, rapid quenching is often desirable. But, in the case of large-scale products quenching or when residual stress has to be controlled, inadequate quenching often occurs. During this process, heterogeneous precipitation takes place and exerts negative effects on the final mechanical properties, i.e., giving rise to lower hardness, strength, corrosion resistance and fracture toughness after aging[4−9]. Hence, many studies have been carried out on the effect of heterogeneous precipitation on the properties of these alloys by step-quenching method[10−12], and time−tempreature−properties diagrams have been gotten. In contrast, less attention has been paid to the heterogeneous precipitation itself[13].

Aluminum alloy 7055 has been developed for upper wing in aircraft due to the combination of high strength, high fracture toughness and high corrosion resistance achieved by T77 heat treatment[14]. In a previous work, the authors evaluated the time−temperature−properties diagrams for this alloy by step-quenching method, and found that this alloy is quite quench sensitive[12]. In this work, the effect of step-quenching on the microstructure of the same alloy after aging was studied so as to improve the understanding of the quench sensitivity and the precipitation behavior during quenching.

2 Experimental

2.5 mm-thick hot-rolled sheet of aluminum alloy 7055 was used. The nominal chemical composition is Al-8.0Zn-1.8Mg-2.3Cu-0.17Zr(mass fraction, %). After solution heat treatment at 470 ℃ for 60 min, samples were transferred to a salt bath within 3 s. The salt bath temperatures were 235 ℃, 355 ℃ and 415 ℃, respectively, which are in the critical temperature range for aluminum alloy 7055[12]. After holding for different time, the samples were further quenched into water at

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room temperature and aged artificially at 121 ℃ for 24 h.

Vickers hardness tests were carried out on the aged samples using Model HV−10B hardmeter. Samples for transmission electron microscopy (TEM) examination were ground to 0.1 mm, punched into *d*3 mm, electro-chemically polished using solution of 70%CH_3OH+30%H_3NO_3 below −20 ℃, and observed on Tecnai G^2 20 transmission electron microscope operated at 200 kV.

3 Results

3.1 Hardness measurements

The effect of holding time at 235 ℃, 355 ℃ and 415 ℃ on the hardness of aluminum alloy 7055 after artificial aging is indicated in Fig.1. With the increase in holding time at 235 ℃, the hardness decreases slowly before 150 s, and rapidly after 150 s. The hardness is lower than HV100 after about 3 000 s. So, the samples held for 60 s, 570 s, 4 140 s are selected for microstructure examination. At 355 ℃, the hardness decreases greatly after holding for only 10 s, and further decline can be observed with prolonging isothermal holding time. After about 360 s, the hardness remains almost the same. So, the samples held for 10 s, 60 s and 360 s are selected for microstructure examination. At 415 ℃, the hardness decreases slowly with holding time. And the samples held for 60 s and 1 800 s are selected for microstructure examination.

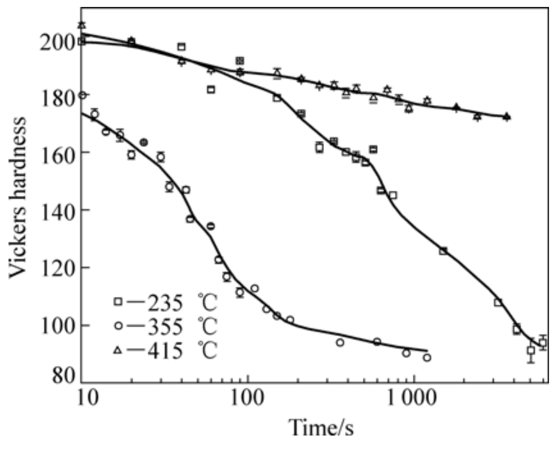

Fig.1 Effect of step-quenching on hardness of aged aluminum alloy 7055

3.2 TEM observation

3.2.1 Effect of holding time at 235 ℃ on microstructure

Fig.2 shows effect of holding time at 235 ℃ on the microstructure of aluminum alloy 7055 after artificial aging. It can be seen that there are many coarse η equilibrium phase particles with size of about 200 nm in the matrix of the sample held for 60 s (Fig.2(a)). These η equilibrium phase particles are plate- or rod-shaped. Higher magnification image shows there are plenty of dispersed fine η' precipitates inside grains, which make contribution to high hardness. The η phase particles at grain boundaries are quite large and close-spaced. The average width of grain boundary precipitates free zone (PFZ) is about 50 nm.

With holding time increasing to 570 s, some parallel thin-rod *S* phases can be seen apart from coarse η equilibrium phase (Fig.2(c)). This was also reported in Ref.[13], in which precipitation of η phase prior to *S* phase during holding at 250 ℃ was observed. It is supposed that during isothermal holding at 235 ℃, η phase precipitates earlier than *S* phase. After aging, precipitates free zones are created around these coarse particles, as shown in Fig.2(d). During artificial aging, the coarse particles continue to grow via absorption of solutes around them in the matrix due to their size advantage, and inhibit nucleation of fine precipitates, leading to PFZ. Moreover, it can be seen that most η equilibrium phase particles are associated with dispersoids, as indicated by arrows in Fig.2(d). The size of these dispersoids is about 40 nm. It is obvious that these dispersoids contain zirconium because zirconium is the only trace element added to the alloy. And formation of equilibrium phase on Zr-containing dispersoids has been observed by many researchers[4, 13, 15]. The coarse equilibrium phase makes little contribution to hardness, but leads to depletion of solutes in the matrix. Consequently, less amount of fine hardening precipitates are formed after aging, leading to lower hardness, as shown in Fig.1. The η phase particles at grain boundaries are quite large and close-spaced. The average width of grain boundary precipitates free zone (PFZ) is increased to about 110 nm.

When the holding time is extended to 4 140 s, the hardness of the aged sample is lowered to HV 95, i.e., little aging hardening effect is achieved. According to TEM images (Figs.2(e) and (f)), the matrix is filled with coarse particles, and very few fine precipitates can be identified. This is probably responsible for the low hardness. Combined with selected area diffraction(SAD) pattern (insert in Fig.2(f)), it is supposed that these coarse particles are mainly made up of η and *S* phases. The η phase particles at most grain boundaries are larger and still close-spaced, and the width of PFZ is significantly increased to about 230 nm.

3.2.2 Effect of holding time at 355 ℃ on microstructure

Fig.3 shows the effect of holding time at 355 ℃ on the microstructure of aluminum alloy 7055 after aging. From Fig.3(a) and corresponding SAD pattern, it can be seen that holding time of only 10 s at this temperature results in many coarse plate-/rod-like η equilibrium phase particles in the matrix. The size of these particles is from 80 nm to 700 nm. According to the contrast

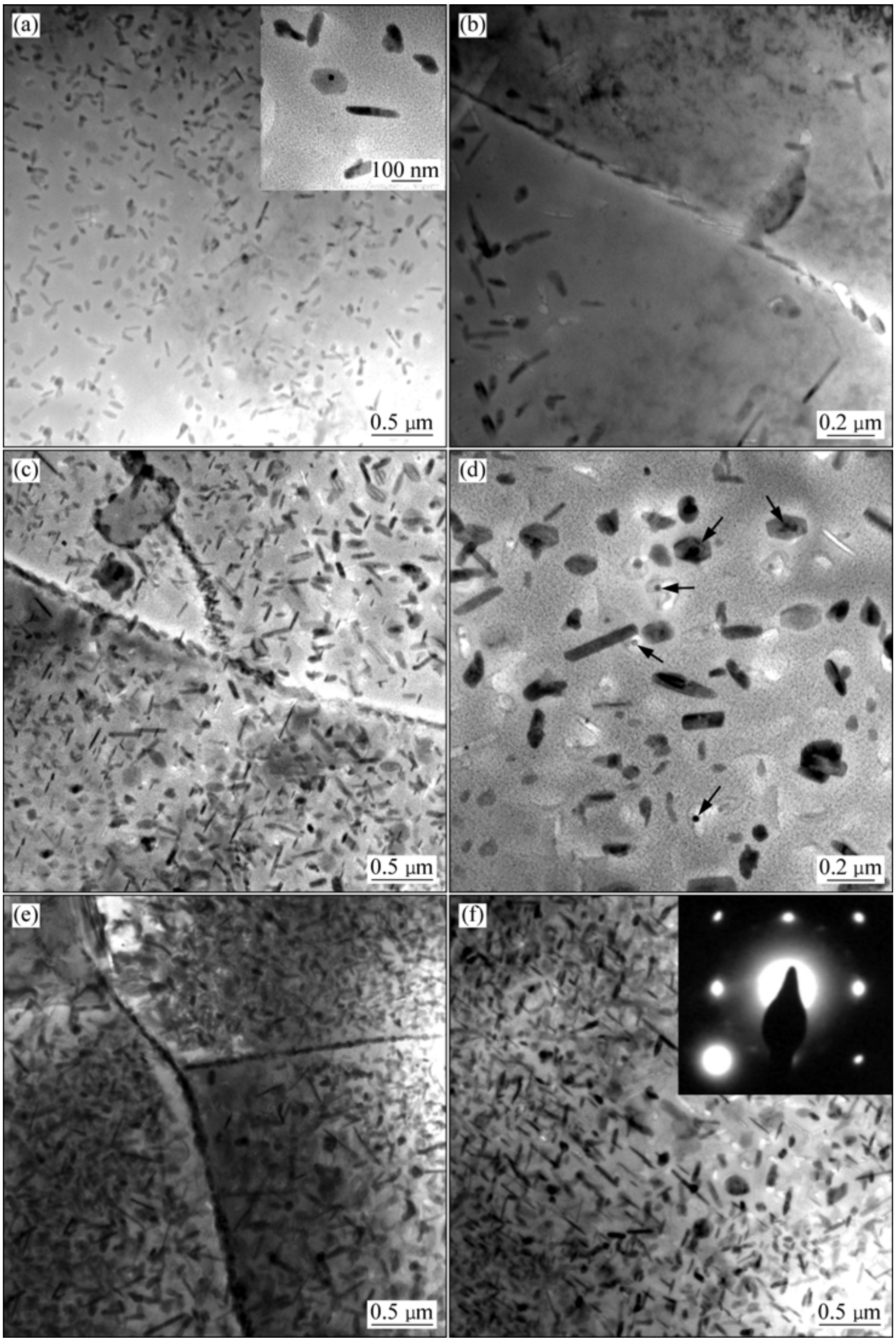

Fig.2 Effect of holding time at 235 ℃ on microstructure of Al alloy 7055 after artificial aging: (a), (b) 60 s; (c), (d) 570 s; (e), (f) 4 140 s (Insert: [001] SAD pattern)

between location near and that far away from these coarse particles, it is evident that precipitates free zones form after aging. There are many dispersed fine η' precipitates inside the grains, so the hardness is still as high as HV170. The η phase particles at grain boundaries become more-spaced with PFZ width of about 60 nm(Fig.3(b)).

When the holding time is extended to 60 s, the number of the coarse η equilibrium phase is increased, and the size is in the range of 100−700 nm. Typical TEM image is shown in Fig.3(c). Among the η equilibrium phase particles, there are a few fine η' precipitates, as shown in Fig.3(d). This kind of microstructure leads to inadequate hardening after aging and lower hardness, as shown in Fig.1. It can also be seen that most η equilibrium phase particles are associated with Zr-containing dispersoids (indicated by arrows in Fig.3(d)), which often act as effective nucleation sites for

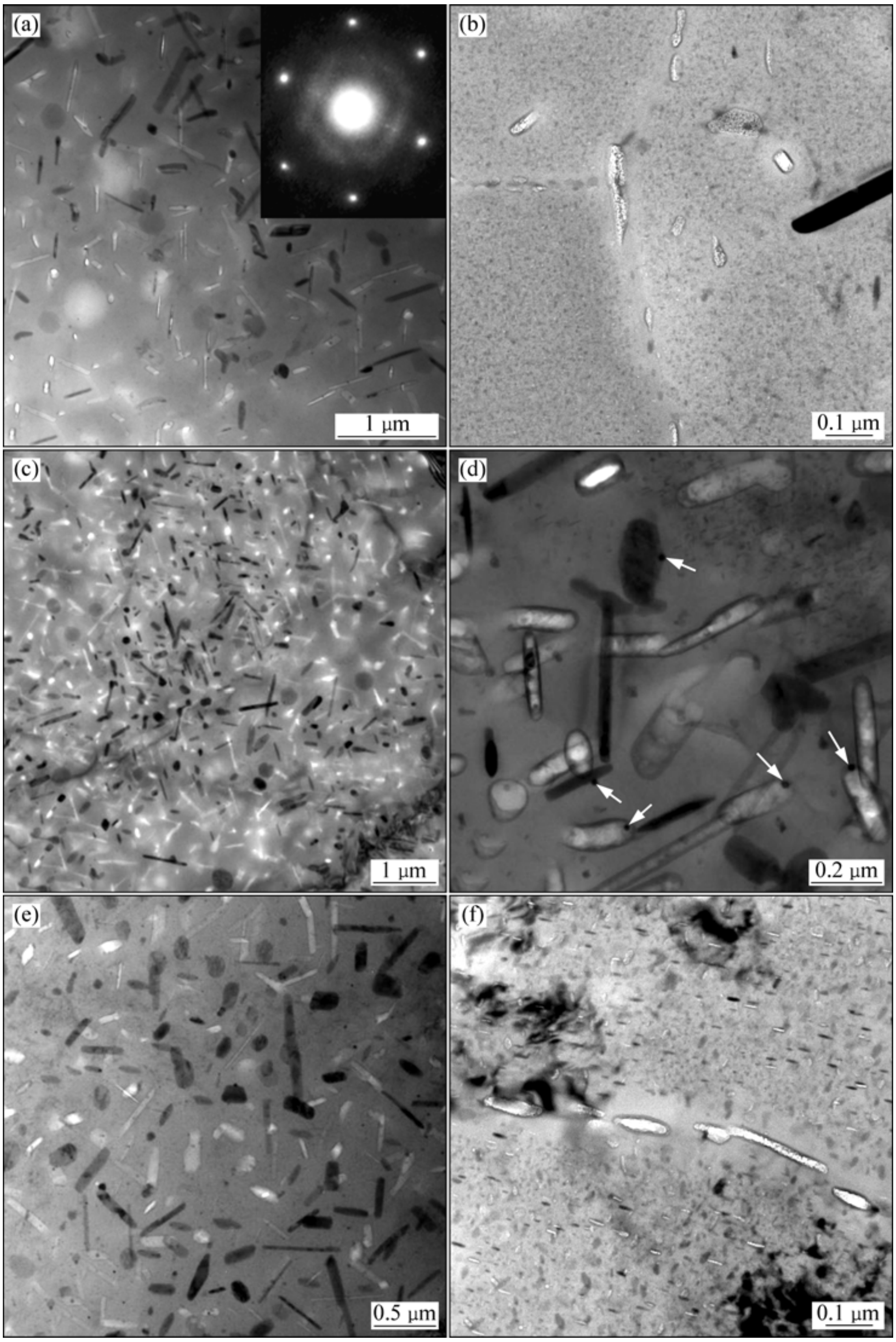

Fig.3 Effect of holding time at 355 ℃ on microstructure of Al alloy 7055 after artificial aging: (a), (b) 10 s (Insert: [111] SAD pattern in matrix); (c), (d) 60 s; (e), (f) 360 s

heterogeneous precipitation[4].

With extension of holding time to 360 s, the number of η equilibrium phase particles is slightly decreased, while their size is increased (Fig.3(e)). During this period, coarsening of η equilibrium phase mainly occurs. Inside grains, some η' hardening precipitates can be observed, but the density is smaller and the size is larger(Fig.3(f)). The η phase particles at grain boundaries are coarse and distributed discontinuously. The average width of grain boundaries PFZ is about 120 nm.

3.2.3 Effect of holding time at 415 ℃ on microstructure

Fig.4 shows the effect of holding time at 415 ℃ on the microstructure of aluminum alloy 7055 after aging. There is a mixture of GP zone and fine η' precipitates in the matrix held for 60 s, so the hardness is quite high. Moreover, many Al_3Zr dispersoids with size of about 40 nm can be observed, as indicated by arrows in Fig.4(a). At most grain boundaries, the η phase particles are quite small and distributed continuously, and the precipitates free zone is quite narrow, as shown in Fig.4(b).

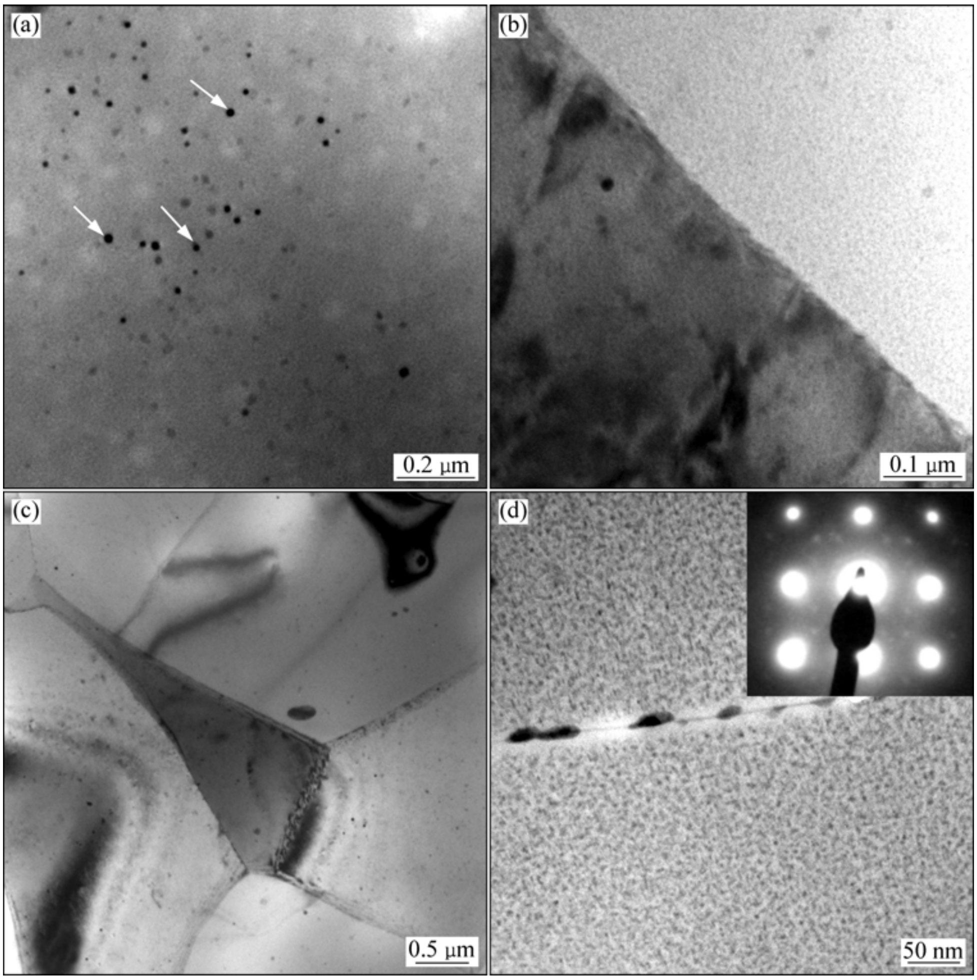

Fig.4 Effect of holding time at 415 ℃ on microstructure of Al alloy 7055 after artificial aging: (a), (b) 60 s; (c), (d) 1 800 s (Insert: [001] SAD pattern in matrix)

When the alloy is held for 1 800 s, few coarse particles can be seen in the matrix, as shown in Fig.4(c). There are plenty of fine η' precipitates in the matrix according to SAD pattern in Fig.4(d), but their size is slightly larger and density is slightly lower, which is probably responsible for lower hardness shown in Fig.1. Another significant change is that η phase particles at most grain boundaries are coarser and more spaced, and the width of precipitates free zone is about 25 nm, as shown in Fig.4(d).

4 Discussion

During isothermal holding at 235−415 ℃, there are a lot of equilibrium phase particles precipitated by heterogeneous nucleation, because homogeneous nucleation is believed to occur only when the holding temperature is below 200 ℃[13]. These heterogeneous nucleation sites primarily consist of grain boundaries and Al_3Zr dispersoids, but their nucleation stimulating efficiency may be influenced by isothermal holding temperature.

According to microstructure shown in Fig.4, few large η equilibrium phase particles can be seen in the matrix of the aged alloy after isothermal holding at 415 ℃ which is only 25 ℃ lower than the solvus temperature of η phase[13]. At this temperature, it seems that no heterogeneous nucleation occurs on Al_3Zr dispersoids, which may be attributed to their nucleation stimulating inefficiency. However, grain boundaries are effective nucleation sites for their high interface energy at high temperature[16]. Due to lower solubility of alloying elements at 415 ℃, the solid solution is decomposed mainly by heterogeneous nucleation because of low driving force. So, equilibrium phase tends to form heterogeneously at grain boundaries; absorbs solutes to grow to a larger size; and simultaneously inhibits nucleation of new precipitates at other part of the grain boundaries during subsequent low temperature aging, thus leading to the more spaced and coarser particles at grain boundaries, as shown in Fig.4(d). With extension of time at this temperature, solutes in the matrix flow to the grain boundaries, leading to lower saturation of solutes after quenching.

Moreover, vacancy concentration is lower due to the low quenching temperature, which may decrease homogeneous nucleation rate during aging[2]. As a result, lower hardness is obtained due to lower density of hardening precipitates with larger size, as shown in Fig.1 and Fig.4.

When the isothermal holding temperatures are lowered to 355 ℃ and 235 ℃, a number of coarse particles can be observed inside grains. Most of these coarse particles are associated with Al_3Zr dispersoids, as indicated by arrows in Fig.2 and Fig.3. It is supposed that Al_3Zr dispersoids start to be effective nucleation sites for equilibrium phase as temperature is decreased. These coarse particles have little hardening effect due to their large size, but give rise to depletion of Zn, Mg solutes in the solid solution. As a result, less amount of hardening precipitates can be formed after aging. Moreover, precipitates free zones are created around these coarse particles. This kind of microstructure makes little contribution to hardening as shown in Fig.1. With extension of holding time, more coarse particles form and grow gradually to a larger size, and thus more solutes are consumed. Consequently, the hardness after aging decreases gradually with isothermal holding time increasing.

After holding for a certain period, the hardness of the aged alloy tends to be a certain value. For instance, the hardness is about HV 130 after holding for about 360 s at 355 ℃ as shown in Fig.1. But, it is not observed for isothermal holding at 415 ℃ and 235 ℃, probably because the holding time at these two temperatures is not long enough to reveal the trend. The contents of Zn, Mg elements in aluminum matrix should reach their solubility limits after enough holding time at a certain temperature, and the excess solutes precipitate in the form of coarse particles. Higher temperature means higher solubility limit of alloying elements in the aluminum matrix. As a result, more amount of solutes in the solid solution available for hardening precipitates can be frozen by quenching following higher temperature holding, thus leading to higher hardness after aging, which is the case shown in Fig.1.

5 Conclusions

1) Isothermal holding at 415 ℃ results in formation of η phase at grain boundaries. Prolonging holding time leads to coarser and more spaced η phase particles at grain boundaries with wider precipitates free zone, and lower density of larger η' hardening precipitates inside grains after aging.

2) Isothermal holding at 355 ℃ and 235 ℃ results in formation of coarse η/S phase on dispersoids and at grain boundaries. Prolonging holding time leads to fewer η' hardening precipitates inside grains, larger and more spaced η phase particles at grain boundaries and wider grain boundary precipitates free zone after aging.

References

[1] LUMLEY R N, POLMEAR I J, MORTON A J. Development of mechanical properties during secondary aging in aluminium alloys [J]. Materials Science and Technology, 2005, 21(9): 1025−1032.

[2] BERG L K, GJΦNNES J, HANSEN V E A. GP-zones in Al-Zn-Mg alloys and their role in artificial aging [J]. Acta Mater, 2001, 49(17): 3443−3451.

[3] WANG Tao, YIN Zhi-ming, SHEN Kai. Single-aging characteristics of 7055 aluminum alloy [J]. Transaction of Nonferrous Metals Society of China, 2007, 17(3): 548−552.

[4] LIU Sheng-dan, ZHANG Xin-ming, CHEN Ming-an. Effect of Zr content on quench sensitivity of AlZnMgCu alloys [J]. Transactions of Nonferrous Metals Society of China, 2007, 17(4): 787−792.

[5] DUMONT D, DESCHAMPS A, BRÉCHET C. Characterization of precipitation microstructures in aluminium alloys 7040 and 7050 and their relationship to mechanical behavior [J]. Materials Science and Technology, 2004, 20(5): 567−576.

[6] MORERE B, EHRSTROM J, GREGSON P J. Microstructural effects on fracture toughness in AA7010 plate [J]. Metallurgical and Materials Transactions A, 2000, 31(10): 2503−2515.

[7] DORWARD R C, BEENSEN D J. Grain structure and quench-rate effects on strength and toughness of AA7050 Al-Zn-Mg-Cu-Zr alloy [J]. Matallurgical Transaction A, 1995, 26(10): 2855−2865.

[8] LIU S D, ZHANG X M, CHEN M A. Influence of aging on quench sensitivity effect of 7055 aluminum alloy [J]. Materials Characterization, 2008, 59(1): 53−60.

[9] ZHANG Xin-ming, LIU Sheng-dan, LIU Ying. Influence of quench rate and zirconium content on intergranular corrosion of 7055 type aluminum alloy [J]. Journal of Central South University: Science and Technology, 2007, 38(2): 181−185. (in Chinese)

[10] STALEY J T. Quench factor analysis of aluminium alloys [J]. Materials Science and Technology, 1987, 3(11): 923−935.

[11] ROBINSON J S, CUDD R L, TANNER D A. Quench sensitivity and tensile property inhomogeneity in 7010 forgings [J]. Journal of Materials Processing Technology, 2001, 119(1/3): 261−267.

[12] LIU Sheng-dan, ZHANG Xin-ming, HUANG Zhen-bao, LIU Wen-hui. Quench sensitivity of 7055 aluminum alloy [J]. Journal of Central South University: Science and Technology, 2006, 37(5): 846−849. (in Chinese)

[13] GODARD D, ARCHAMBAULT P, AEBY-GAUTIER E. Precipitation sequences during quenching of the AA7010 alloy [J]. Acta Materialia, 2002, 50(9): 2319−2329.

[14] WILLIAMS J C, STARKE J E A. Progress in structural materials for aerospace systems [J]. Acta Materialia, 2003, 51(19): 5775−5799.

[15] DESCHAMPS A, BRÉCHET C. Influence of quench and heating rates on the ageing response of an Al-Zn-Mg-(Zr) alloy [J]. Materials Science and Engineering A, 1998, 251(1/2): 200−207.

[16] PORTER D A, EASTERLING K E. Phase transformation in metals and alloys [M]. Oxford, Britain: Alden Press, 1981.

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